IS 4524 (1989): Acetoacet-o-chloroanilide [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard

(Reaffirmed 2000)

ACETOACET-*O*-CHLOROANILIDE — SPECIFICATION

(First Revision)

भारतीय मानक

एसीटोऐसेट-ओ-क्लोरोएनिलाइड — विशिष्टि

(पहला पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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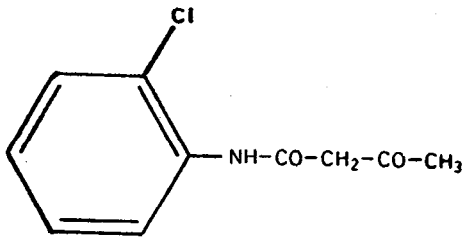
Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 20 October 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Acetoacet-*o*-chloroanilide ($C_{10}H_{10}Cl\ NO_2$) is an important dye intermediate used in the manufacture of Yellow organic pigments. It has the following structural formula:



Acetoacet-*o*-Chloroanilide
(Molecular Mass 211·6)

CAS Registry Number [93-70-9]

This standard was first issued in 1968. The Committee responsible for its preparation decided to revise it in order to introduce chromatographic method for estimation of *o*-Chloroaniline. The requirement of melting range has also been modified.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ACETOACET-*O*-CHLOROANILIDE — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for acetoacet-*o*-chloroanilide.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
915 : 1975	One-mark graduated flasks (*first revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
2552 : 1979	Steel drums (galvanized and ungalvanized) (*second revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white powder.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Acetoacet-*o*-Chloroanilide

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex A
(1)	(2)	(3)	(4)
i)	Melting range	103 to 105°C	A-1
ii)	Matter insoluble in sodium hydroxide solution, percent by mass, *Max*	0·2	A-2
iii)	o-Chloroaniline, percent by mass, *Max*	0·2	A-3
iv)	Assay, percent by mass, *Min*	98	A-4

4 PACKING AND MARKING

4.1 Packing

Unless otherwise agreed, the material shall be suitably packed in wooden barrels, multi-walled paper sacks, or suitable drums (*see* IS 2552 : 1979).

4.2 Marking

The containers shall be securely closed and shall be marked legibly and indelibly with the following information:

a) Name of the material;

b) Indication of the source of manufacture;

c) Batch number; and

d) Net, gross and tare mass.

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 The representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Assay and the tests for the determination of melting range shall be conducted on each of the individual samples separately.

5.2.2 Test for the determination of matter insoluble in sodium hydroxide solution and o-Chloroaniline content shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay and melting range, if each of the individual test results as obtained in **5.2.1** satisfies the corresponding requirement given in Table 1.

5.3.2 *For Composite Samples*

The lot shall be declared as conforming to the requirement of matter insoluble in sodium hydroxide solution and o-Chloroaniline content, if the test result as obtained in **5.2.2** satisfy the corresponding requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out as prescribed in the appropriate clauses of Annex A indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1, and *Clause* 6.1)

METHODS OF TEST FOR ACETOACET-*O*-CHLOROANILIDE

A-1 DETERMINATION OF MELTING RANGE

A-1.1 Determine the melting range of the material as given in 8 of IS 5299 : 1969.

A-2 MATTER INSOLUBLE IN SODIUM HYDROXIDE SOLUTION

A-2.1 Reagent

A-2.1.1 *Sodium Hydroxide Solution*, 1 N.

A-2.2 Procedure

Take 10 ml of the sodium hydroxide solution in 100-ml volumetric flask and add 1 g of the material. Shake well and allow the solution to stand for 5 minutes. Make up the volume with water. Filter through a sintered crucible of porosity G4, wash thoroughly, dry, weigh and calculate the percent matter insoluble in alkali.

A-2.3 Calculation

$$\text{Matter insoluble in sodium hydroxide solution, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of material taken for the test.

A-3 DETERMINATION OF *O*-CHLOROANILINE

A-3.0 General

Thin layer chromatographic method is employed for estimation of *o*-chloroaniline.

A-3.1 Apparatus

A-3.1.1 *Thin Layer Chromatographic Plate*

Glass plate of size 10 × 20 cm, coated uniformly with silica gel. G of thickness of 250 micron and activated at 110°C for 30 minutes.

A-3.1.2 *Micropipette*, 10 µl capacity.

A-3.1.3 *Developing Chamber*

Suitable reactangular glass jar which contains the developer and which is closed well with the lid for saturation.

A-3.2 Reagents

A-3.2.1 *Methyl Alcohol*

A-3.2.2 *Fast Blue B Salt* (*Tetrazotised-o-Dianisidine Zinc Double Salt*), 1·0 percent solution in water.

A-3.2.3 *American Base* [*N-(1-Naphthyl) Ethylenediamine Dihydrochloride*], 0·1 percent solution in methanol.

A-3.2.4 *Eluent*, a mixture of butyl acetate and acetic acid (99 : 1).

A-3.3 Procedure

A-3.3.1 *Preparation of Sample Solution*

Dissolve 0·2 g (on 100 percent basis) of sample in 5 ml methanol (4·0 percent solution).

A-3.3.2 *Preparation of Standard Solution*

Prepare (standard) solution as above. Prepare 0·004 percent solution of *ortho*chloroaniline in methanol.

A-3.3.3 Spot 10 µl each of standard and sample solution on the first plate. Spot 10 µl each of standard solution, sample solution and *ortho*chloroaniline solution on the second plate. Allow to dry. Then place the plate in the solvent of the developing chamber in a vertical manner and close chamber. Allow to run for 13 cm. Take out the plates and dry the solvent completely. Spray the first plate with Fast Blue B salt solution (**A-3.2.2**). Compare the intensity of the spot visually with that of the known standard (yellow single spot).

Diazotise the second plate by putting it into a chamber containing nitrous fumes for 1 minute and then spray the plate with American Base solution (A-3.2.3). Compare the intensity of the spot with the spot of known concentration of *ortho*chloroaniline for both standard as well as sample. The maximum allowable limit for *ortho*-chloroaniline in standard as well as sample is 0·1 percent.

A-4 ASSAY

A-4.0 Outline of the Method

A standard diazonium solution is prepared by reacting *o*-chloroaniline with standard sodium nitrite solution. The sample of the *acetoacet-o-chloroanilide* is then titrated with the standard diazonium solution.

A-4.1 Reagents

A-4.1.1 *Hydrochloric Acid*, 30 percent (*m/v*).

A-4.1.2 *Standard Sodium Nitrite Solution*, 1 N, freshly standardized.

A-4.1.3 p-*Chloroaniline*, 99 percent pure.

A-4.1.4 *Sodium Hydroxide Solution*, 40 percent (*m/v*).

A-4.1.5 *Dilute Acetic Acid*, 25 percent (*m/v*).

A-4.1.6 *Pyridine* (*Water White Grade*), 90°/160°C.

A-4.1.7 *H-Acid Indicator Solution*, dissolve 0·5 g H-Acid in 100 ml water containing 1 g of soda ash.

A-4.1.8 *Tetrazodianisidine Solution* — Take 2 g of dianisidine base in a beaker and dissolve in 7 ml of hydrochloric acid (heat, if necessary, up to 50°C). Cool with ice to about 0°C and add immediately 12 ml of sodium nitrite solution and make up the volume to 100 ml with ice cold water. Shake mildly. Test for excess sodium nitrite with starch iodide paper. Store this solution in an amber coloured bottle in a cool place.

A-4.1.9 *Starch-Iodide Test Papers*

A-4.2 Procedure

A-4.2.1 *Preparation of Standard Diazonium Solution of* p-*Chloroaniline*

Take about 4 g of pure *p*-chloroaniline in a 250-ml glass beaker containing 10 ml of hydrochloric acid and 150 ml of water, heat up to 60°C to dissolve completely. Cool externally to 0 to 5°C with chopped ice, titrate with standard sodium nitrite solution with gentle stirring. Test the solution for excess of sodium nitrite by spotting on starch-iodide test paper. The end point is reached when an immediate faint blue coloured ring appears on starch-iodide test paper which may be obtained repeatedly during a period of 10 minutes without further addition of sodium nitrite solution. Allow to stand for 30 minutes. Filter to remove insoluble matter, then make up the filtrate to 250 ml in a volumetric flask. Store the standard diazonium solution in ice-bath in the dark.

A-4.2.2 Weigh accurately about 0·780 g of (acetoacet-*o*-chloroanilide test sample and dissolve with 1 ml of sodium hydroxide in 25 ml of water, stir this solution and add dropwise dilute acetic acid till the *p*H is 7 which may be judged by using *p*H paper. Dissolve under good stirring precipitated acetoacet-*o*-chloroanilide with 25 ml pyridine and few drops of methanol to get a clear solution. While stirring mechanically, add the standard diazonium solution (*see* A-4.2.1) from a burette equipped with a water jacket through which water is circulating at about 10°C. The burette should be of amber glass to minimize any decomposition of the diazonium salt by light. Titrate as rapidly as spot test permits. To test for excess acetoacet-*o*-chloroanilide, place a few drops of titration mixture on a piece of filter paper. About 1 cm away from the edge of the liquid mark, place a few drops of tetrazodianisidine solution. Where the two liquid portions meet on the filter paper, a brown colour will develop, if excess of acetoacet-*o*-chloroanilide is present. Similarly try with H-acid indicator to test the excess of diazonium salt. If a pink colour develops at the interjunction, excess of diazonium solution is indicated.

A-4.2.3 Initially, add the standard diazonium solution in 1 to 2 ml portions, testing titration mixture after each addition for excess of acetoacet-*o*-chloroanilide and diazonium salt. As the end-point approaches, standard diazonium solution should be added in portions of 0·2 ml. The end-point is reached when no reddish brown colour is given with tetrazodianisidine solution and no pink colour or very faint pink colour is given with H-acid indicator. Note the volume of standard diazonium solution required for titration.

A-4.3 Calculation

$$\text{Assay, percent by mass (calculated on molecular mass 211·6)} = \frac{V \times 21{\cdot}16 \times N}{M}$$

where

V = volume in ml of standard diazonium solution required;

N = normality of standard diazonium solution; and

M = mass in g of the sample taken for the test.

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	331 01 31 331 13 75
Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43
Southern : C.I.T. Campus, 4 Cross Road, MADRAS 600113	235 02 16
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) BOMBAY 400093	632 92 95

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